ADMINISTRATIVE INFORMATION

1. **Project Name:** Inverse Process Analysis for the Acquisition of Thermophysical Data

2. **Lead Organization:** University of Tennessee
Mechanical, Aerospace and Biomedical Engineering Department
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3. **Principal Investigator:** Dr. Jay Frankel
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4. **Project Partners:** Oak Ridge National Laboratory
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Supporting Companies: Howmet, Inc., Ford Motor, Co., Flow Science, Inc., ProCAST, Inc., NETZSCH Instruments, Inc.

5. **Date Project Initiated:** 10/01/2001

6. **Expected Completion Date:** 10/01/2004

PROJECT RATIONALE AND STRATEGY

7. **Project Objective:** The overall goal is to increase the accuracy of thermophysical property data, solid fraction and density, for solidification processing. Accurate correlations between the measured data and sample temperature will be developed for Differential Scanning Calorimetry (DSC) and dilatometer instruments. Improved thermophysical property data for materials processing will benefit the aluminum, steel, and metalcasting industries.

8. **Technical Barrier(s) Being Addressed**: Due to instrument time lags, incorrect and inconsistent high-temperature thermophysical properties are often measured.
   - Currently, a method does not exist for properly accounting for the effect of thermal lags in the measurement systems.
   - Inverse modeling techniques for these types of systems do not exist.

9. **Project Pathway:** The pathway for addressing the technical barriers involves four major efforts: (1) heat transfer analysis of the measurement process for determining the time-temperature dependence of instrument parameters, and, (2) calibration of instruments and preparation of operating procedures that incorporate the use of heat transfer models, (3) modifications to the specimen test section of the apparatus, and (4) development of robust models that enable replication of property measurement for other materials.

10. **Critical Technical Metrics:** With current systems, the estimated error for the solid fraction obtained from DSC data is approximately ÷30%. Currently, the dilatometer temperature error is approximately 6°C at temperatures of 600-700 °C. This project aims to obtain solid fraction data with less than ÷3% error and reduce the temperature error during density measurements to ÷0.5 °C.

PROJECT PLANS AND PROGRESS

11. Past Accomplishments:

a. *Analytical Model for DSC and Dilatometer*:

i. **DSC**- A heat transfer model comprising the key components of the DSC system has been developed using time constants to account for thermal lags in the system (ORNL).

ii. **DSC** - *Heat transfer models for instrument asymmetry were successfully validated against experimental results*.

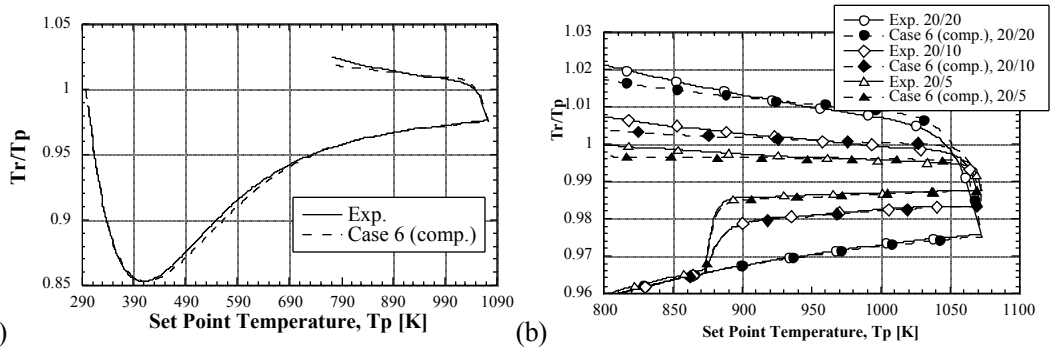

Figure 1. Ratio between the reference plate temperature and set point temperature. The DSC model accurately reproduces the experimental DSC data for different heating and cooling rates.

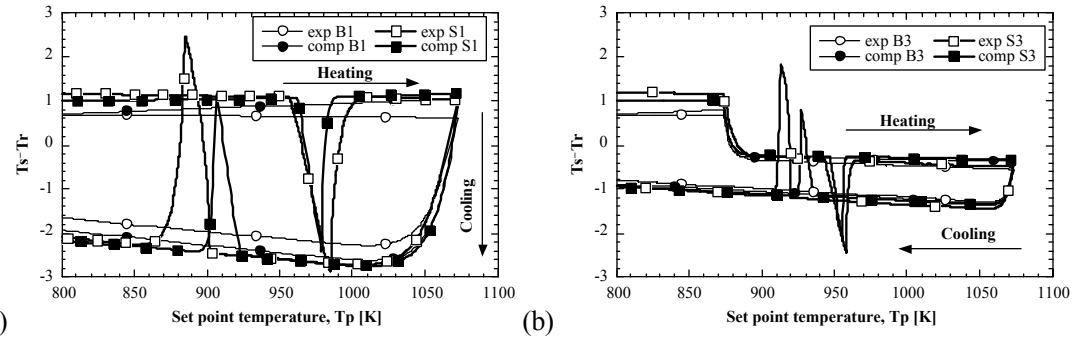

Figure 2. Temperature difference between the sample plate and reference plate for the high temperature domain [800:1100] K: (a) 20 °C/min at heating and cooling and (b) the heating rate changes from 20 to 5°C/min at 873K. Cooling starts at 1073K.

iii. **DSC**-The governing equations for the heat transfer model (1.i) were recast as a parameter estimation problem for inverse analysis (UTK).

iv. **Dilatometer**-A heat flux based inverse heat conduction model was developed for the newly designed specimen test section. A one-input temperature model was also developed for the dilatometer (UTK).

b. *Experimental*:

v. **DSC**- Experiments, in which the heating rate is changed, successfully guided the model development (ORNL). Experimental results show a high degree of reproducibility for various heating and cooling rates.

vi. **Dilatometer**- A sample holder was redesigned to assure symmetry and uniform heating in the sample (ORNL). Using the new design reduces the time to perform an experiment by a factor of ten.

vii. **Dilatometer-** For the graphite specimen test section, the accuracy of the temperature measurements was ÷0.5°C for pure aluminum.

c. *Model Validation*:

i. **DSC heat transfer model has been validated for pure aluminum and alloy A356 (ORNL).**

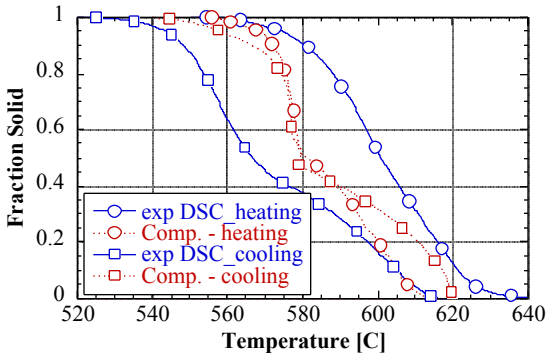

Figure 3. The computed fraction solid for alloy A356 using the current DSC model.

ii. **Dilatometer-** Data is being collected for a ternary aluminum alloy at various heating rates for use in the new inverse model. This data will be used as input to the new inverse model.

12. Future Plans: Quarters 11 and 12 of the three year project will involve completion of the verification process and the required reporting of the program results.

i. **DSC** – (a) **Develop a user friendly DSC model,** (b) install the first software version of the DSC model on a laboratory system, and (c) meet with instrument manufacturers to highlight developments and discuss implementation avenues.

ii. **Dilatometer-** (a) Make a test section from a low thermal conductivity material, (b) Conduct heat-flux based inverse analysis using the new test section, and (c) Complete experimental validation of the inverse analysis approach (August 04) – (UTK).

iii. Reports (Qtr 11 and final) and papers (dilatometer, DSC) are to be completed and submitted to archival journals.

13. Project Changes: An alternative direct approach to the inverse method was developed in order to determine the enthalpy and ensuing distribution of the solid fraction during solidification. This direct approach has proven to be very reliable and efficient technique for performing and analyzing DSC data.

For the dilatometer graphite specimen test section, the temperature gradient in the sample holder precludes the use of inverse methods based on heat flux. Thus, a new model is being developed involving an alternative inverse analysis that uses one thermocouple data stream.

14. Commercialization Potential, Plans, and Activities:
The results of this program will be shared with DSC and dilatometer instrument manufacturers. During meetings and site visits presentations will be made related to the design in the specimen test sections and post processing of the data.

All computational and experimental procedures will also be published and presented at conferences organized by professional associations. The companies supporting this project (Howmet, Inc., Ford

Motor, Co., Flow Science, Inc., ProCAST, Inc., NETZSCH Instruments, Inc.) will have access to the new thermophysical property data generated in this program. ProCAST is distributing with its software a material property database, disseminating the information across industries. The supporting companies will be able to utilize the improved data in their software models.

15. Patents, Publications, Presentations:

Frankel, J.I., M. Keyhani, and K. Taira "A Modified Least-Squares Method for Optimal Solutions in Inverse Problems", *40th AIAA Aerospace Sciences Meeting and Exhibit*, Reno, NV, January 14-17, 2002 (AIAA 2002-0657).

Taira, K., J.I. Frankel, and M. Keyhani, "Metric Analysis for the Modified Discrete Least-Squares Method", *40th AIAA Aerospace Sciences Meeting and Exhibit*, Reno, NV, January 14-17, 2002 (AIAA 2002-0658).

Frankel, J.I., M. Keyhani, K. Taira, "In-Phase Error Estimation of Experimental Data and Optimal First Derivatives", *AIAA Journal*, vol. 42, #5, 2004 (in press).

Frankel, J.I. and K. Taira, "The Use of Parameter Identification in Error Estimation", *22th IASTED International Conference on Modeling, Identification and Control* (MIC 2003), Innsbruck, Austria, February 10-13, 2003.

Osborne, G.E., J.I. Frankel, and A. Sabau, "A New Parameter Estimation Method for DSC Thermodynamic Property Evaluation, Part I: Analytic Developments", *22th IASTED International Conference on Modeling, Identification and Control* (MIC 2003), Innsbruck, Austria, February 10-13, 2003.

Osborne, G.E., J.I. Frankel, and A. Sabau, "A New Parameter Estimation Method for DSC Thermodynamic Property Evaluation, Part II: Runge-Kutta Implementation and Numerical Results", *22th IASTED International Conference on Modeling, Identification and Control* (MIC 2003), Innsbruck, Austria, February 10-13, 2003.

Frankel, J.I. and G.E. Osborne, "The Prediction of Heating/Cooling Rates in Material Science Investigations, *4th International Conference on Quenching and Control of Distortion*, Beijing, China, Nov. 23-25, 2003.

Sabau, A.S., W.D. Porter, and J.I. Frankel, "Conduction and Radiation Parameters for Analytical Models of Differential Scanning Calorimetry Instruments", TMS 2004, Charlotte, NC, March 12-14, 2004.

Osborne, G.E., J.I. Frankel, and A.S. Sabau, "Characterization of Thermal Lags and Resistances in a Heat-Flux DSC", *TMS 2004*, Charlotte, NC, March 12-14, 2004.

Sabau, A.S, and W.D Porter, "Analytical Models for System Errors of Differential Scanning Calorimetery Instruments", 2004 ASME Heat Transfer/Fluids Engineering Summer Conference, Charlotte, NC, July 11-15, 2004 (submitted).